

NEW PROCESS FOR PRODUCING LOW DP MICROCRYSTALLINE CELLULOSE

FIELD OF THE INVENTION

5

The present invention relates to a process for the production of microcrystalline cellulose.

DESCRIPTION OF PRIOR ART

10

Canadian application No. CA 2,313,261 (JOLLEZ) describes a process for the production of microcrystalline cellulose. In this process, the pulp obtained at the end of a thermo mechanical pulping step is submitted to a sudden and violent depressurisation and a shear force. This step results in the production of a non-selective fragmentation of the microcrystalline cellulose, which leads to the production of impurities by the oxidation during and after the explosion of the pulp.

15

Canadian patent No. CA 1,198,703 (DELONG) describes a process which generates a mixture of sugar and cellulose more or less degraded. This process uses wood as starting material and sulphuric acid, sulphur dioxide (SO₂) or hydrochloric acid.

20

Canadian patent No. CA 2,137,890 (BERGFELD) describes a process for converting cellulose fibers derived from a conventional process, into microcrystalline cellulose by using benign reactives like O₂ and CO₂. More particularly, it shows that a low degree of polymerisation can be obtained by the application of high-pressure at 140° to 180°C for 15 minutes to 5 hours on aqueous suspensions of cellulose (solid / liquid ratio of 1/8 to 1/20) in the presence of O₂ and CO₂ in autoclaves in non-continuous mode.

25
30

EXPRESS MAIL CERTIFICATE

Date

11/2/01

Label No.

4767727845US

I hereby certify that, on the date indicated above, this paper or fee was deposited with the U.S. Postal Service & that it was addressed for delivery to the Assistant Commissioner for Patents, Washington, DC 20231 by "Express Mail Post Office to Addressee" service.

Name (Print)

Signature

SUMMARY OF THE INVENTION

5 A first object of the present invention is to provide a process for the manufacture of microcrystalline cellulose having a fibrous appearance and an integrity, which has been kept.

10 A second object of the present invention is to provide a process for the production of microcrystalline cellulose that does not necessitate the use of any mineral acids, sulphur dioxide or carbon dioxide.

A third object of the present invention is to provide a process for the production of microcrystalline cellulose in the absence of violent non-selective depressurisation.

15 The process according to the invention allows the application of a controlled depressurisation, which limits the production of non-desirable derivatives, which in turn allows a high yield of microcrystalline cellulose.

More precisely, the process according to the present invention comprises the following steps:

- 20
- a) preparing a pulp by repulping,
 - b) pressing the pulp obtained in a),
 - c) decompacting the pulp obtained in b),
 - d) feeding the pulp obtained in c) into a pre-heated reactor,
 - 25 e) cooking the pulp with the pre-heated reactor at a temperature, a time and a pressure selected to obtain a pulp having a desired degree of polymerisation,
 - f) cooling said pulp obtained in e) and partially depressurising the reactor by injecting water into said reactor,
 - g) filtering the pulp obtained in f),
 - 30 h) bleaching of the pulp obtained in g), and
 - i) drying the pulp obtained in h).

One of the advantages of this process is that there is no disorganised destruction of the cell structure such as it occurs during a violent depressurisation in the processes using a thermo-mechanical pulping step. In fact, contrary to the thermo-mechanical pulping processes, there is no exposure of the burst material to air, light or hot metallic sides in the process of the present invention. Thus, there is no formation, or very limited formation of oxycellulose or non-desired functionalisation. Indeed, such formation is favoured, in thermo-mechanical processes, by the contact of the fibers to air and metals at the flashing temperature.

Another advantage of the process of the invention is that the filtration of the treated product is much faster, thanks to the absence of fine fragments resulting from the random and non-selective breaking of the cellulose chains during the violent depressurisation, which occurs during the thermo-mechanical treatments like steam explosion treatment.

A further advantage of the process of the invention is that controlled depressurisation prevents disorganised destruction of the cell and gives high yield of microcrystalline cellulose.

These higher yields explain the decrease of the suspended solids and dissolved pollutants in the water phase by more than half compared to a thermo-mechanical pulping process. The decrease is due to the absence of non-selective fragmentation in the process according to the invention and the absence of products of decomposition, which are generated by oxidation during and after the explosion in a thermo-mechanical pulping process.

The process of the invention also has the advantage of allowing more efficient brightening or bleaching. Such is due to the absence of fines resulting from the random breaking of the cells in a conventional steam treatment which retain the impurities and consume much more bleaching reactives. Under such conditions,

the yield of operation is superior to 99% and the peroxide brightens the pulp without delignifying or contributing to the purification of the surrounding impure environment, like in the case of explosive treatments. The degree of brightness of the bleached final product is much higher than in any other treatment by thermo-
5 mechanical pulping.

Another advantage of the process of the present invention is that it is carried out in a low acidity environment. The advantages of such low acidity lies in that it does not cause a massive depolymerization of the cellulose like in the case of the
10 DELONG patent who works with wood and ends up with cellulose that has been cut in a non-selective fashion thereby, giving a mix of sugars and fragments of cellulose chains in the presence of numerous degradation products like furfural and other products coming from hemicelluloses or lignin.

15 The present invention and its advantages will be more easily understood after reading the following non-restrictive description of the preferred embodiments thereof, made with reference to the hereinbelow drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

20 Figure 1 is a photographic representation of a Temalfa cellulose 93 TEM prior to being processed.

25 Figure 2 is a photographic representation of a Temalfa cellulose of Figure 1, treated by a steam explosion process.

Figure 3 is a photographic representation of the Temalfa cellulose of Figure 1, treated by the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

As mentioned hereinabove, the process of the invention comprises the steps of:

5

- a) preparing a pulp by repulping,
- b) pressing the pulp obtained in a),
- c) decompacting the pulp obtained in b),
- d) feeding the pulp obtained in c) into a pre-heated reactor,
- 10 e) cooking the pulp at a temperature, a time and a pressure allowing to obtain a pulp having a desired degree of polymerisation,
- f) cooling and partially depressurising the reactor injecting water into the reactor,
- g) filtering the pulp obtained in f),
- h) bleaching of the pulp obtained in g), and
- 15 i) drying the pulp obtained in h).

20

During the cooking process at high temperature, the lignocellulosic material undergoes controlled hydrolysis. The hydrolysis can be accelerated or slowed down by the presence of acids or bases during the cooking. At the same time, an oxidation can take place if the environment is favourable.

25

Under the effect of the temperature and the acidity of the reaction medium, an hydrolysis of hemicelluloses and lignin, if there are any left, can take place along with the hydrolysis of the amorphous zones of the cellulose. This hydrolysis will be more or less severe depending on the raw material, on the aqueous environment and obviously on the conditions of pressure, time and temperature applied during the treatment.

30

The hydrolysis can take place thanks to the organic acids such as acetic acid, freed by the thermal rupture of the acetyl groups of the hemicelluloses chains. Such organic acids may serve as catalysts for the hydrolysis of other products, notably cellulose.

This phenomenon is illustrated by the fact that the pH during steam cooking, goes rapidly from about 4.5 to 3.5 depending on the type of the pulp. A kraft pulp from softwood, for an equal treatment, will give a lower pH than a sulphite pulp from softwood, because of the higher content of hemicelluloses in the kraft pulp. Obviously, the extent of such effect depends on the severity of the applied treatment.

Oxidation of the product present in the process can take place with more or less intensity depending on the time of exposure to air, the temperature, the environment and the accessibility to the treated product. This oxidation will lead to degradation of products hence, to a cellulose product of lower quality than desired as well as lower yields.

The non-controlled oxidation can also give coloured products. It may also degrade or alter the product resulting in the production of oxycelluloses for example.

Types of celluloses that can be treated by the process of the present invention.

The cellulose employed in the process of the present invention may be derived from a wide variety of cellulosic feedstock including but not limited to, wood and wood products, such as wood pulp fibres, non-woody paper-making fibres, from cotton, from straws and grasses, such as rice and esparto, from canes and reeds such as bagasse, from bamboos, from stalks with fibres, such as jute, flax, kenaf, cannabis, linen and ramie, and from leaf fibres such as abaca and sisal.

Suitable wood sources include softwood sources such as pines, spruces and firs, and hardwood sources such as oaks, eucalyptuses, poplars, beeches and aspens.

Bleached, partially bleached or non bleached celluloses from resinous or hardwoods, and resulting from chemical processes such as kraft process or sulphite as well as cellulose resulting from alternative processes such as steam

explosion treatment may also be used.

Types of additives that can be used with the present process.

- 5 Any suitable antioxidant may be used for the purpose of the present invention. More particularly, any other product having antioxidant function and that is acceptable with the desired applications of the finish products and compatible with the operation conditions may be used.
- 10 Preferably, these antioxidants are selected from the group consisting of:
 - Propyl gallate,
 - Hydroquinone,
 - Sodium sulfite, and
 - 15 - Citric acid.

Commercial products such as EDTA and Dequest from Monsanto may also be used in the process of the present invention.

20 Steps of the process

The pulp used as the starting material of the process of the present invention is prepared by repulping the cellulose in water in the presence or absence of an additive, antioxidant or sequestrant, in a reactor mixed with the recirculation pump
 25 working at a 2% to 3% consistency

The repulped pulp is pumped towards a pressing system such as a screw press or any other device allowing to drain and to lower the moisture of the fibre to 70% or less in weight (wet basis).

30

The humid pulp is then decompacted and aerated on a shredder or a coarse grinder. The reactor is then pre-heated to the temperature desired or to any other

temperature chosen to reduce the condensation due to the heating of the walls during the treatment. This is done via the jacket or by injecting vapour directly and then emptying it before opening it to charge it.

- 5 The cooking reactor is then fed with wet grounded pulp. In continuous mode, the feeding is done through an airlock or by any other mechanism allowing to feed a vessel that is under pressure for example a co-axial system. The reactor is then closed when the apparatus in question works in batch mode. Vacuum can be applied before the steam feed to purge the gases present, such as air.

10

The reactor is then fed with steam directly up to a predetermined pressure. This method allows to rapidly reach a temperature between 200° and 235°C.

15

A purge of non-condensables, through the top of the reactor, in the case of a batch reactor is desirable if the purge was not carried out. Furthermore, steam must be re-introduced in the reactor to maintain the pressure.

20

The cooking is maintained during 4 to 25 minutes depending on the nature of the cellulose and the chosen working temperature. The goal is to reach a stable degree of polymerisation indicative of reaching the desired DP for MCC.

25

In a batch mode, the reactor is then rapidly cooled by an injection of water in the jacket and in the reactor itself. A preliminary depressurisation of the excess vapour can also be carried out before the injection of cooling water.

30

In a continuous mode, the treated product is pushed to one or several partially decompressed chambers for partial decompression. This insures the transport of the product towards the exit, without causing any explosion. The product is thereafter cooled down by water injection and further transported for the next step.

A variant of the decompression chambers may be carried out by means of a set of screw spindles and/or gears and/or inverted pump. This variant insures a rapid

cooling of the product by a partial decompression with no explosion of the latter.

According to a preferred embodiment of the present invention, the cooking and cooling steps are carried out as follows. The humid cellulose cooks under
5 pressurized water vapor (330 psi for example) and is transported into the cooking reactor via a screw press.

The cooking (or hydrolysis) lasts, for example, 15 minutes at a temperature of 220°C. The cooking time is determined by the rotation of the screw, which pushes
10 the cellulose through the cooking reactor. When the cellulose reaches the other end of the cooking reactor, it is emptied into a vertical container, which contains water. This water is pumped into the vertical container by a pump capable of overcoming the pressure inside the cooking reactor.

15 The pressure of the water to be added into the vertical container must be equal or greater than 330 psi in order to penetrate into the system. When the liquid level inside the vertical container reaches the high level indicator, the water pump stops, and the lower valve (discharge valve) located under the container is opened. At this point, the vapor present inside the cooking reactor forces the liquid (water +
20 cellulose) out of the system. This occurs without any vapor loss, because when the water-cellulose mixture reaches the low-level indicator of the vertical container, the output (discharge) valve is closed again.

The discharged liquid and cellulose go from a pressure of 330 psi (inside the
25 container) to atmospheric pressure without vaporization of water, because the temperature of the water does not exceed 80°C.

There is only one release (discharge) step which is responsible for a pressure drop of almost 330 psi. There is thus, no vaporization of the water contained in the
30 cellulose, which produces a random bursting of cellulose, as there is in a process using a thermo-mechanical pulping step.

In the past, the random bursting of cellulose was unavoidable because it was necessary to make the water-containing cellulose go from 220°C at 330 psi to 100°C at atmospheric pressure in a split second. After the closing of the discharge valve, the cycle begins all over again with the injection of water into the vertical container up to the high level mark.

In the present invention, the product is quickly depressurized by mixing it with water so as to avoid bursting of the cellulose, all the while maintaining an uninterrupted cooking in the cooking reactor. The cooking reactor is not affected by what is going on in the vertical container, because it never undergoes any no drop in temperature or pressure.

The water which one injects does consume a small amount of vapor since it passes from 20°C to 80°C, all the while making it possible to isolate the cooking reactor from the atmospheric pressure. The discharge is done in cycles, while the cooking reactor works in a continuous mode (uninterrupted) for the hydrolysis.

The mixing can then start and the reactor is cooled down to around 60°C by adding water to recuperate all the cellulose present in the reactor.

When the treated pulp is a pulp of sulphite or bleached kraft quality, it is sent directly to filtration before going to "brightening" and/or bleaching.

When the pulp is of intermediate quality, it may be treated with a caustic soda solution that is diluted in a way to eliminate leftover lignin and other impurities present. Then, it is filtered and washed before being sent to bleaching, which will be done according to the initial quality of the starting cellulose.

After filtration, the product (from Temalfa TEM 93 and Domtar Q90 cellulose) is brightened with hydrogen peroxide in the following conditions:

Peroxide: 2% w/w on dry mass;

Magnesium sulphate: 0.5% w/w on dry mass; and

Sodium hydroxide: 0.5% w/w on dry mass.

- 5 The treatment can be carried out at a temperature ranging between 60 and 120°C under air or oxygen pressure reaching up to 120 psi.

10 The brightening and bleaching process can be adapted in function of the quality of the initial product, and in the more extreme cases, known bleaching methods can be used, such as hypochlorite or chlorine dioxide bleaching. The bleaching consistency will preferably be 25% but this can also be done at lower consistencies.

15 The bleached pulp is filtered and may be used as such or in a dry state for new applications comprising a new generation of microcrystalline cellulose of fibrous appearance, but having the same specifications as a classical microcrystalline cellulose in crystallinity index and DP.

20 The filtered bleached pulp can also be homogenised in water at a consistency going from 0.5 to preferably 3% and then filtered and washed to rid the residue of bleaching reactives. The pH of the solution, if needed, may be adjusted with hydrochloric acid (HCl) or ammonium hydroxide (NH₄OH) so to obtain a pH ranging between 5.5 to 7. This operation is done with an apparatus of the "blender" type or colloid mill, which allows the separation of microcrystalline
25 cellulose particles to give non-colloidal microcrystalline cellulose.

After filtration, the suspension obtained is brought to a dryer of the type "spray dryer" to obtain the size required in the desired dryness of classical microcrystalline cellulose, for instance at a consistency of 10 to 20%.

Results yield of MCC obtained by the process of the invention.

	ALPHA 93 ¹	ALPHA 93 ²	KRAFT ¹	KRAFT ²
Repulping	100	100	100	100
Hydrolysis and washing	95,0	87,3	88,0	83,2
H ₂ O ₂ and washing	99,0	88,9	99,0	93,3
NaOCl and washing (if needed)			99,0	89,0
Homogenization	99,5		98,5	
Drying	99,5	99,5	99,0	99,0
Total Yield	93,1	77,2	84,1	68,4

- 5 **ALPHA 93¹** : results obtained from the process of the present invention
ALPHA 93² : results obtained from a process using a thermo-mechanical pulping step
KRAFT¹ : results obtained from the process of the present invention
KRAFT² : results obtained from a process using a thermo-mechanical pulping step

10 There is an increase in the yield of the alpha-pulp of 20% and an increase in the yield of the kraft pulp of 23% compared to the thermo-mechanical pulping process using steam explosion treatment.

- 15 Figures 1 to 3 clearly shows the difference in composition of the microcrystalline resulting from a process using a thermo-mechanical pulping step versus one resulting from the process of the present invention.

20 Applications of the microcrystalline cellulose obtained by the process of the present invention.

The bleached product that went through the blender and that was spray dried, has similar applications as the classical applications for MCC PH 101 (microcrystalline

cellulose sold by FMC under the trademark AVICELL) , that is for instance:

- Tableting (excipient with bonding properties);
- Cream used in pharmaceuticals and cosmetics;
- 5 • Fat replacer (lipid free ice cream and mayonnaise);
- Chromatography support; and
- Complexation with transition metals for enzyme immobilisation.

10 The microcrystalline cellulose obtained by the process of the present invention may be used for different applications. Indeed the process of the present invention allows the production of a microcrystalline cellulose having fibrous characteristics.

15 This cellulose is of very high purity and serves as a support for a new type of catalysts.

20 Since the structure of the product has a fibrous aspect and that, contrary to classical MCC, OH groups from the anhydroglucose molecule are not available, they will not react with the metals used to obtain a catalyst. Furthermore, in mixing this preparation with inorganic products for a sufficient mixing and drying time, the distribution of the active sites formed then dried and charred, will be different than the one obtained with a classical microcrystalline cellulose conferring new properties to the finished product. The spherical substrate of the catalyst, after charring, contains holes of controlled dimension making it different than the one obtained with colloidal MCC or with ground cellulose, which is, on top of that,
25 limited by its initial inferior quality.

Particularities of the process according to the invention for obtaining microcrystalline cellulose developed by Kemestrie.

30 The present section details the particularities of the process of the present invention. It summarises the various characteristics of the process of the present invention that renders it different from the one that are already known.

- Steam cooking of humidified cellulose that is saturated in water.

- Cooking without any mineral acids or dioxides.

5

- Presence or absence of additives (e.g. antioxidant).

- No explosion of the treated product.

- 10 - It is applicable to many types of cellulose of deciduous or resinous trees.

- Cooking of the humidified cellulose with saturated steam.

15

- Controlled cooking allowing to obtain the desired degree of polymerization of the cellulose.

- Very short time of treatment thanks to the instantaneous heating of the cellulose with saturated steam.

20

- Limited vapour consumption that is 1 to 1,2 ton of vapour per ton of dry cellulose.

25

- Contrary to the thermo-mechanical pulping, this new process prevents exposure of burst material to air, to light, or to the hot metallic sides. Therefor, there is no possible or very little formation of oxycelluloses, which is favoured in the presence of metals at these temperatures. Moreover, we know that when the substance is subjected to violent depressurisation such as going from 350 psi to atmosphere pressure in a few fractions of second, such as in the case of thermo-mechanical pulping, the substance is treated in a destructive fashion. This also has an abrasive effect on the material of the reactor located near the exit, thus increasing the chance for the treated product to be contaminated with metallic particles.

30

- The addition of certain cooking additives can help to avoid even more oxidation

of the cellulose and its impurities.

- Very low formation of colour on the treated product with the recommended process.

5

- Increased efficiency of washing (which means reduction of water quantities used).

10

- A degree of brightness of the finished bleached product higher than any other treatment by steam explosion.

15

- If need be, a homogenisation of the finished product can be carried out and the breaking of the cellulose chains is done in a methodical manner contrary to what is done by classical thermo-mechanical pulping with the random explosion of cells as well as with the shear and the impact produced by the violent depressurisation.

20

- More precisely, with the alpha 93 pulp the yield of the initial dry pulp is 95% at the hydrolysis including the washing whereas with an explosive process where in the best of the cases as disclosed in patent no. CA 2,313,261 this yield is at best of 87% under similar conditions.

- With kraft pulp, the yield under similar conditions is of 88% versus 83% by steam explosion treatment.

25 EXAMPLES

- A) TEMALFA 93 cellulose: small scale test without additives
- B) TEMALFA 93 cellulose: small scale test with additives
- C) Kraft cellulose: small scale test without additives
- 30 D) TEMALFA 93 on a commercial scale without additives.

Temalfa 93 cellulose from Tembec Company is obtained by the sulfite process

from resinous trees. Given its quality, its standards of whiteness, its purity and its low content in resin, this pulp can be easily used in the production of carboxymethyl cellulose, of methyl cellulose and of microcrystalline cellulose (MCC) for the grades 100 or 200. This pulp is characterised in that it gives a degree of polymerisation of the MCC in the vicinity of 225.

Temalfa 93 is the most commonly used feedstock around the world for the fabrication of microcrystalline cellulose in classical processes using mineral acid.

10 The composition of the Temalfa cellulose is the following:

Pentosans:	2.40%
Ashes:	0.05%
S10 at 25 C:	8.6%
15 S8 at 25 C:	5.6%
Alpha cellulose	92,5%

The kraft cellulose from Donohue at 100% resinous has the following composition:

20 Pentosans:	7.00%
Ashes:	0.36%
Alpha cellulose:	89%.

Domtar Q90 pulp may also be used in the context of the present invention.

25

Brightness: 90

Viscosity : 22

Shives: 0 PPM

Impurities: 0.4 PPM

30 Moisture: 10%

Alpha cellulose >90%

A - example 1: Temalfa 93 cellulose

1kg of Temalfa 93 cellulose was repulped at a consistency of 2.5% in water, then partially dried with the help of a press and coarsely grounded to obtain a residual
5 moisture of 60.3%.

From the above-obtained product, 229 g (equivalent to 90.913 g of cellulose) were introduced in a 24 litres reactor pre-heated with saturated steam. The steam is then introduced directly from the bottom of the reactor and a rapid purge is carried
10 out to evacuate the non condensables.

Within 1 minute the product reached a temperature of 220°C where it is maintained for 13 minutes. The pressure is then partially released and pressurised cold water is injected in the reactor in such a way as to allow rapid cooling of the
15 pulp. Mixing is initiated at this stage to ensure a homogeneous discharge and to carry on to the next step of the treatment. The washed filtered product (252 g at 65.7% moisture) is white, slightly greyish.

The pH of the filtered solution is 5.3.
20

Using a sample of 59,7 g a brightening with hydrogen peroxide was carried out with 2% peroxide in the presence of 0.5% magnesium sulphate (on a dry pulp basis) at a pH of 10.5. The operation was carried out for 1 hour at 60°C.

25 After filtration and washing, 56.7 g of pulp is recovered (64.2% moisture).

A homogenisation of 55.7 g of brightened pulp gives, after filtration and washing, 50.7 g of pulp at 60.8% moisture (19,9 g of dry product).

30 ANALYSIS:

DP (Degree of Polymerisation) = 214

Cr.I (Cristallinity Index) = 84,6

MS (Microcrystal Size) = 46,6 Å

B - Example 2: Temalfa 93 cellulose with additives.

A solution of 1% sodium sulphite is used at a ratio of 20/1 on 100 g of Temalfa cellulose. After pressing and coarse grinding, 214 g of soaked cellulose at 75.3% moisture is introduced into the pre-heated reactor.

The product is treated as in the example 1 for 12 minutes. After filtration and washing, 363 g of pulp at 75.3% moisture is obtained and the pH of the filtrate is 4.3.

357 g of bleached pulp obtained above is brightened with peroxide at the same conditions as in example 1. After washing and filtration, 253.3 g of pulp is recovered (moisture = 65.5%).

A homogenisation is carried out with 250 g of brightened pulp described above and after filtration and washing, 237.7 g of pulp is recovered (64% moisture).

ANALYSIS:

DP = 219

Cr.I = 88,9

MS = 46,6 Å

C - Example 3: kraft cellulose

210 g of kraft cellulose humidified at 55.8% is treated at 220°C for 13 minutes.

After filtration and washing, 366.4 g of cellulose are recovered at 77.7% moisture. The pH of the filtered solution is 4. The cellulose obtained is coloured, light

brown/caramel.

A brightening step is carried out with the same conditions as previously described. A bleaching step is then carried out with hypochlorite with 1% hypochlorite (on dry
5 cellulose basis) at a pH of 11 at 40°C during 2 hours. The filtered bleached product has a weight of 237.5 g and a humidity of 66.2%. The homogenisation allowed the recovery of 240.4 g of pulp at 67.1% humidity.

ANALYSIS:

10

DP = 224

Cr.I = 88,8

MS = 43,1 Å

15 D - Example 4: Example at a Commercial Scale

120 kg of Temalfa 93 cellulose was repulped in the reactor mixed with cold water at a consistency of 3%. The operation is done in 6 steps of repulping of 20 kg each.

20

The pulp is then sent to a screw press of Atara/Spirac Spiropress U-260 brand to be dried up to a residual humidity of approximately 65%. The wet cellulose obtained goes through a moulding granulator that will decompact it.

25 The product obtained is loaded in a cylindrical stainless steel reactor. The reactor's volume is 2 cubic meters. After having closed the reactor, it is directly fed with steam to obtain the pressure required for the treatment. In just a few minutes the temperature into the reactor reaches 220°C.

30 After 12 minutes of cooking at 220°C, water is injected in the reactor in order to lower the temperature rapidly and allow a discharge of the cooking product. The discharge of the reactor is done several times with water injection to allow for a

complete recuperation of the product.

4 cubic meters of water are required to complete this operation.

- 5 A rotating filter of 0.9 meter diameter and 0.6 meter length is then used for the filtration and the washing of the cellulose that is obtained.

The product has a fibrous aspect, reflecting from a non-destructive process. It is whitish.

10

ANALYSIS:

DP = 214

Cr.I = 85,2

MS = 46,6 Å.

15

E- Example 5: Microcrystalline cellulose manufacture in continuous mode

20 kg of Q 90 Domtar pulp was re-pulped at a consistency of 3% in water, than partially dried with the help of a press and coarsely ground to obtain residual moisture of 64%.

20

The reactor is heated up to 220° by direct steam injection and the rate of the screw is determined to have a residence time of 16 minutes.

- 25 The moist cellulose is fed to the hopper during 6 hours accordingly with the opening cycle of the ball valves. The cooked product is exits the reactor accordingly with water cycle. At the same time, water is injected into the vessel above the reactor. When the water reaches predetermined level into the vessel the ball valves opens and closes without loss of steam through the valve.

30

The product is then filtered on rotary filter and the sequence of washing and bleaching with hydrogen peroxide continues. After adjustment to pH 6.5 with

ammonium hydroxide the microcrystalline cellulose is finally homogenised into a colloid mill and then dried into a commercial spray drier in order to give an average powder of 50 microns.

- 5 This MCC has a DP of 222, a bulk density of 0,29 and pass the ID and the compaction tests comparing with Avicell 101 standard.

Although the present invention has been explained hereinabove by way of a preferred embodiment thereof, it should be pointed out that any modifications to
10 this preferred embodiment within the scope of the present description is not done to alter or change the nature and scope of the present invention.

11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65
66
67
68
69
70
71
72
73
74
75
76
77
78
79
80
81
82
83
84
85
86
87
88
89
90
91
92
93
94
95
96
97
98
99
100
101
102
103
104
105
106
107
108
109
110
111
112
113
114
115
116
117
118
119
120
121
122
123
124
125
126
127
128
129
130
131
132
133
134
135
136
137
138
139
140
141
142
143
144
145
146
147
148
149
150
151
152
153
154
155
156
157
158
159
160
161
162
163
164
165
166
167
168
169
170
171
172
173
174
175
176
177
178
179
180
181
182
183
184
185
186
187
188
189
190
191
192
193
194
195
196
197
198
199
200
201
202
203
204
205
206
207
208
209
210
211
212
213
214
215
216
217
218
219
220
221
222
223
224
225
226
227
228
229
230
231
232
233
234
235
236
237
238
239
240
241
242
243
244
245
246
247
248
249
250
251
252
253
254
255
256
257
258
259
260
261
262
263
264
265
266
267
268
269
270
271
272
273
274
275
276
277
278
279
280
281
282
283
284
285
286
287
288
289
290
291
292
293
294
295
296
297
298
299
300
301
302
303
304
305
306
307
308
309
310
311
312
313
314
315
316
317
318
319
320
321
322
323
324
325
326
327
328
329
330
331
332
333
334
335
336
337
338
339
340
341
342
343
344
345
346
347
348
349
350
351
352
353
354
355
356
357
358
359
360
361
362
363
364
365
366
367
368
369
370
371
372
373
374
375
376
377
378
379
380
381
382
383
384
385
386
387
388
389
390
391
392
393
394
395
396
397
398
399
400
401
402
403
404
405
406
407
408
409
410
411
412
413
414
415
416
417
418
419
420
421
422
423
424
425
426
427
428
429
430
431
432
433
434
435
436
437
438
439
440
441
442
443
444
445
446
447
448
449
450
451
452
453
454
455
456
457
458
459
460
461
462
463
464
465
466
467
468
469
470
471
472
473
474
475
476
477
478
479
480
481
482
483
484
485
486
487
488
489
490
491
492
493
494
495
496
497
498
499
500
501
502
503
504
505
506
507
508
509
510
511
512
513
514
515
516
517
518
519
520
521
522
523
524
525
526
527
528
529
530
531
532
533
534
535
536
537
538
539
540
541
542
543
544
545
546
547
548
549
550
551
552
553
554
555
556
557
558
559
560
561
562
563
564
565
566
567
568
569
570
571
572
573
574
575
576
577
578
579
580
581
582
583
584
585
586
587
588
589
590
591
592
593
594
595
596
597
598
599
600
601
602
603
604
605
606
607
608
609
610
611
612
613
614
615
616
617
618
619
620
621
622
623
624
625
626
627
628
629
630
631
632
633
634
635
636
637
638
639
640
641
642
643
644
645
646
647
648
649
650
651
652
653
654
655
656
657
658
659
660
661
662
663
664
665
666
667
668
669
670
671
672
673
674
675
676
677
678
679
680
681
682
683
684
685
686
687
688
689
690
691
692
693
694
695
696
697
698
699
700
701
702
703
704
705
706
707
708
709
710
711
712
713
714
715
716
717
718
719
720
721
722
723
724
725
726
727
728
729
730
731
732
733
734
735
736
737
738
739
740
741
742
743
744
745
746
747
748
749
750
751
752
753
754
755
756
757
758
759
760
761
762
763
764
765
766
767
768
769
770
771
772
773
774
775
776
777
778
779
780
781
782
783
784
785
786
787
788
789
790
791
792
793
794
795
796
797
798
799
800
801
802
803
804
805
806
807
808
809
810
811
812
813
814
815
816
817
818
819
820
821
822
823
824
825
826
827
828
829
830
831
832
833
834
835
836
837
838
839
840
841
842
843
844
845
846
847
848
849
850
851
852
853
854
855
856
857
858
859
860
861
862
863
864
865
866
867
868
869
870
871
872
873
874
875
876
877
878
879
880
881
882
883
884
885
886
887
888
889
890
891
892
893
894
895
896
897
898
899
900
901
902
903
904
905
906
907
908
909
910
911
912
913
914
915
916
917
918
919
920
921
922
923
924
925
926
927
928
929
930
931
932
933
934
935
936
937
938
939
940
941
942
943
944
945
946
947
948
949
950
951
952
953
954
955
956
957
958
959
960
961
962
963
964
965
966
967
968
969
970
971
972
973
974
975
976
977
978
979
980
981
982
983
984
985
986
987
988
989
990
991
992
993
994
995
996
997
998
999
1000
1001
1002
1003
1004
1005
1006
1007
1008
1009
1010
1011
1012
1013
1014
1015
1016
1017
1018
1019
1020
1021
1022
1023
1024
1025
1026
1027
1028
1029
1030
1031
1032
1033
1034
1035
1036
1037
1038
1039
1040
1041
1042
1043
1044
1045
1046
1047
1048
1049
1050
1051
1052
1053
1054
1055
1056
1057
1058
1059
1060
1061
1062
1063
1064
1065
1066
1067
1068
1069
1070
1071
1072
1073
1074
1075
1076
1077
1078
1079
1080
1081
1082
1083
1084
1085
1086
1087
1088
1089
1090
1091
1092
1093
1094
1095
1096
1097
1098
1099
1100
1101
1102
1103
1104
1105
1106
1107
1108
1109
1110
1111
1112
1113
1114
1115
1116
1117
1118
1119
1120
1121
1122
1123
1124
1125
1126
1127
1128
1129
1130
1131
1132
1133
1134
1135
1136
1137
1138
1139
1140
1141
1142
1143
1144
1145
1146
1147
1148
1149
1150
1151
1152
1153
1154
1155
1156
1157
1158
1159
1160
1161
1162
1163
1164
1165
1166
1167
1168
1169
1170
1171
1172
1173
1174
1175
1176
1177
1178
1179
1180
1181
1182
1183
1184
1185
1186
1187
1188
1189
1190
1191
1192
1193
1194
1195
1196
1197
1198
1199
1200
1201
1202
1203
1204
1205
1206
1207
1208
1209
1210
1211
1212
1213
1214
1215
1216
1217
1218
1219
1220
1221
1222
1223
1224
1225
1226
1227
1228
1229
1230
1231
1232
1233
1234
1235
1236
1237
1238
1239
1240
1241
1242
1243
1244
1245
1246
1247
1248
1249
1250
1251
1252
1253
1254
1255
1256
1257
1258
1259
1260
1261
1262
1263
1264
1265
1266
1267
1268
1269
1270
1271
1272
1273
1274
1275
1276
1277
1278
1279
1280
1281
1282
1283
1284
1285
1286
1287
1288
1289
1290
1291
1292
1293
1294
1295
1296
1297
1298
1299
1300
1301
1302
1303
1304
1305
1306
1307
1308
1309
1310
1311
1312
1313
1314
1315
1316
1317
1318
1319
1320
1321
1322
1323
1324
1325
1326
1327
1328
1329
1330
1331
1332
1333
1334
1335
1336
1337
1338
1339
1340
1341
1342
1343
1344
1345
1346
1347
1348
1349
1350
1351
1352
1353
1354
1355
1356
1357
1358
1359
1360
1361
1362
1363
1364
1365
1366
1367
1368
1369
1370
1371
1372
1373
1374
1375
1376
1377
1378
1379
1380
1381
1382
1383
1384
1385
1386
1387
1388
1389
1390
1391
1392
1393
1394
1395
1396
1397
1398
1399
1400
1401
1402
1403
1404
1405
1406
1407
1408
1409
1410
1411
1412
1413
1414
1415
1416
1417
1418
1419
1420
1421
1422
1423
1424
1425
1426
1427
1428
1429
1430
1431
1432
1433
1434
1435
1436
1437
1438
1439
1440
1441
1442
1443
1444
1445
1446
1447
1448
1449
1450
1451
1452
1453
1454
1455
1456
1457
1458
1459
1460
1461
1462
1463
1464
1465
1466
1467
1468
1469
1470
1471
1472
1473
1474
1475
1476
1477
1478
1479
1480
1481
1482
1483
1484
1485
1486
1487
1488
1489
1490
1491
1492
1493
1494
1495
1496
1497
1498
1499
1500
1501
1502
1503
1504
1505
1506
1507
1508
1509
1510
1511
1512
1513
1514
1515
1516
1517
1518
1519
1520
1521
1522
1523
1524
1525
1526
1527
1528
1529
1530
1531
1532
1533
1534
1535
1536
1537
1538
1539
1540
1541
1542
1543
1544
1545
1546
1547
1548
1549
1550
1551
1552
1553
1554
1555
1556
1557
1558
1559
1560
1561
1562
1563
1564
1565
1566
1567
1568
1569
1570
1571
1572
1573
1574
1575
1576
1577
1578
1579
1580
1581
1582
1583
1584
1585
1586
1587
1588
1589
1590
1591
1592
1593
1594
1595
1596
1597
1598
1599
1600
1601
1602
1603
1604
1605
1606
1607
1608
1609
1610
1611
1612
1613
1614
1615
1616
1617
1618
1619
1620
1621
1622
1623
1624
1625
1626
1627
1628
1629
1630
1631
1632
1633
1634
1635
1636
1637
1638
1639
1640
1641
1642
1643
1644
1645
1646
1647
1648
1649
1650
1651
1652
1653
1654
1655
1656
1657
1658
1659
1660
1661
1662
1663
1664
1665
1666
1667
1668
1669
1670
1671
1672
1673
1674
1675
1676
1677
1678
1679
1680
1681
1682
1683
1684
1685
1686
1687
1688
1689
1690
1691
1692
1693
1694
1695
1696
1697
1698
1699
1700
1701
1702
1703
1704
1705
1706
1707
1708
1709
1710
1711
1712
1713
1714
1715
1716
1717
1718
1719
1720
1721
1722
1723
1724
1725
1726
1727
1728
1729
1730
1731
1732
1733
1734
1735
1736
1737
1738
1739
1740
1741
1742
1743
1744
1745
1746
1747
1748
1749
1750
1751
1752
1753
1754
1755
1756
1757
1758
1759
1760
1761
1762
1763
1764
1765
1766
1767
1768
1769
1770
1771
1772
1773
1774
1775
1776
1777
1778
1779
1780
1781
1782
1783
1784
1785
1786
1787
1788
1789
1790
1791
1792
1793
1794
1795
1796
1797
1798
1799
1800
1801
1802
1803
1804
1805
1806
1807
1808
1809
1810
1811
1812
1813
1814
1815
1816
1817
1818
1819
1820
1821
1822
1823
1824
1825
1826
1827
1828
1829
1830
1831
1832
1833
1834
1835
1836
1837
1838
1839
1840
1841
1842
1843
1844
1845
1846
1847
1848
1849
1850
1851
1852
1853
1854
1855
1856
1857
1858
1859
1860
1861
1862
1863
1864
1865
1866
1867
1868
1869
1870
1871
1872
1873
1874
1875
1876
1877
1878
1879
1880
1881
1882
1883
1884
1885
1886
1887
1888
1889
1890
1891
1892
1893
1894
1895
1896
1897
1898
1899
1900
1901
1902
1903
1904
1905
1906
1907
1908
1909
1910
1911
1912
1913
1914
1915
1916
1917
1918
1919
1920
1921
1922
1923
1924
1925
1926
1927
1928
1929
1930
1931
1932
1933
1934
1935
1936
1937
1938
1939
1940
1941
1942
1943
1944
1945
1946
1947
1948
1949
1950
1951
1952
1953
1954
1955
1956
1957
1958
1959
1960
1961
1962
1963
1964
1965
1966
1967
1968
1969
1970
1971
1972
1973
1974
1975
1976
1977
1978
1979
1980
1981
1982
1983
1984
1985
1986
1987
1988
1989
1990
1991
1992
1993
1994
1995
1996
1997
1998
1999
2000
2001
2002
2003
2004
2005
2006
2007
2008
2009
2010
2011
2012
2013
2014
2015
2016
2017
2018
2019
2020
2021
2022
2023
2024
2025
2026
2027
2028
2029
2030
2031
2032
2033
2034
2035
2036
2037
2038
2039
2040
2041
2042
2043
2044
2045
2046
2047
2048
2049
2050
2051
2052
2053
2054
2055
2056
2057
2058
2059
2060
2061
2062
2063
2064
2065
2066
2067
2068
2069
2070
2071
2072
2073
2074
2075
2076
2077
2078
2079
2080
2081
2082
2083
2084
2085
2086
2087
2088
2089
2090
2091
2092
2093
2094
2095
2096
2097
2098
2099
2100
2101
2102
2103
2104
2105
2106
2107
2108
2109
2110
2111
2112
2113
2114
2115
2116
2117
2118
2119
2120
2121
2122
2123
2124
2125
2126
2127
2128
2129
2130
2131
2132
2133
2134
2135
2136
2137
2138
2139
2140
2141
2142
2143
2144
2145
2146
2147
2148
2149
2150
2151
2152
2153
2154
2155
2156
2157
2158
2159
2160
2161
2162
2163
2164
2165
2166
2167
2168
2169
2170
2171
2172
2173
2174
2175
2176
2177
2178
2179
2180
2181
2182
2183
2184
2185
2186
2187
2188
2189
2190
2191
2192
2193
2194
2195
2196
2197
2198
2199
2200
2201
2202
2203
220